

## **Final Report: (NAG9-1006) Genesis Noble Gas Measurements**

### **Introduction**

The original thrust of our Genesis funding was to extend and refine the noble gas analytical capabilities of this laboratory to improve the precision and accuracy of noble gas measurements in order to optimize the scientific return from the Genesis Mission. This process involved both instrumental improvement (supplemented by a SRLIDAP instrument grant) and refinement of technique. The Genesis landing mishap shifted our emphasis to the irregular aluminum heat shield material from the flat collector wafers. This has required redesign of our laser extraction cells to accommodate the longer focal lengths required for laser extraction from non-flat surfaces. Extraction of noble gases from solid aluminum surfaces, rather than thin coatings on transparent substrates has required refinement of controlled-depth laser ablation techniques. Both of these bring new problems, both with potentially higher blanks from larger laser cells and the larger quantities of evaporated aluminum which can coat the sapphire entrance ports. This is mainly a problem for the heavy noble gases where larger extraction areas are required, necessitating the new aluminum vapor containment techniques described below.

With the Genesis Mission came three new multiple multiplier noble gas mass spectrometers to this laboratory, one built solely by us (Supergnome-M), one built in collaboration with Nu-Instruments (Noblesse), and one built in collaboration with GVI (Helix). All of these have multiple multiplier detection sections with the Nu-Instruments using a pair of electrostatic quad lenses for isotope spacing and the other two using mechanically adjustable positions for the electron multipliers. The Supergnome-M and Noblesse are installed and running. The GVI instrument was delivered a year late (in March 2005) and is yet to be installed by GVI. As with all new instruments there were some initial development issues, some of which are still outstanding. The most serious of these are performance issues with the miniature channel electron multipliers. The delayed installation of Helix by the GVI is partly due to failure of the initial batch of Burle channel multipliers to perform as expected. A number of the channel multipliers designed for Noblesse by Burle have also failed upon baking. Burle has now refined the design of these and we have installed two of the new multipliers and are assessing their performance. The remaining multipliers will be upgraded to the new design from Burle once we confirm that the problem has been fixed.

Measurements of heavy noble gases from Genesis samples with Noblesse are expected as soon as the new extraction cell is coupled with that instrument. Initial measurements of the light noble gases with Supergnome-II, our existing single multiplier have been completed and reported at the Gordon Research Conference, Genesis session, in June 2005. Accuracy of the reported isotopic ratios are currently limited to about one percent (as determined by calibration standards of the same size), while the precision with which we can measure isotopic ratios with this same sample size is two orders of magnitude greater. In order to capitalize on the available precision, we have undertaken an extensive program to evaluate the things that degrade our precision. With the GS-61 ion source space charge effects can distort the extraction field inside of the ionization region. As the space charge increases due to the presence of many ions, the field gradients change slightly reducing the

extraction efficiency and hence the sensitivity. In addition, with space charge distorting the extraction fields, ions stay in the ionization region longer, increasing the chance to become doubly-charged. Therefore, the quantity of ions in the mass spectrometer has an impact on both the effective neon sensitivity (extraction efficiency) and the accuracy of the measured isotopic ratios. The distorted extraction fields causes changes, and hence uncertainties, in the doubly-charged isobaric interferences ( $^{40}\text{Ar}^{++}$  on  $^{20}\text{Ne}$  and  $44^{++}(\text{CO}_2)$  on  $^{22}\text{Ne}$ ) that must be corrected for, thus limiting the accuracy of neon isotopic ratios. To obtain the highest precision and accuracy for solar wind neon, the large quantities of solar wind helium, which contributes to this problem, must be removed. We have built a liquid helium activated charcoal cold finger to trap the solar wind neon while the excess solar wind helium is pumped away.

In addition to solving the space charge problem, we have also addressed the problem of variable apparent mass discrimination which currently limits our neon isotopic accuracy to one percent. We have built two totally new and independent ion counting systems to assess and address the problem with count-rate dependent discrimination. One of these is based upon a counting system with a substantially greater dynamic range than is delivered by the Stanford Research Systems SR400 previously used by us in ion counting. The second system has a pulse discrimination that is based upon the total charge delivered per pulse, instead of the pulse height itself which is the conventional way to do pulse discrimination in ion counting which can be more sensitive to noise and pulse reflections. However, this system does have a substantially longer dead time (about 50 ns, compared with  $< 10$  ns for the current ion counting system). Dead time can be accurately corrected for as long as it remains constant. The first tests of these new counting systems have been completed. Increasing the dynamic range of the counting system apparently does little to improve the count-rate variable mass discrimination we have observed. Within the next few days we will have tested the charge sensitive detection system.

Our existing mass spectrometers, designed and constructed by our own group, are optimized for ion transmission, capable of counting every noble gas atom that is ionized, thus providing the ultimate sensitivity for noble gases. However, these can count only one isotope at a time, meaning that, for xenon with its 9 isotopes, we must throw away 8/9 of the Xe extracted from each sample. The only significant improvement for this loss is the simultaneous detection of multiple isotopes, the common thrust of each of these new Genesis instruments. Complications occur because: 1) Each noble gas has different isotope spacing, and 2) The small electron multipliers required for multiple multiplier detector systems have very limited dynamic range and counting efficiencies less than 100%. These issues are addressed differently in each of the three mass spectrometers we have built and each of the instruments is optimized for a specific task. The Nu-Instruments mass spectrometer utilizes a novel electronic zoom lens to correct for the different isotope spacing for each noble gas and 7 independent multipliers. The GVI instrument utilizes a 120 degree magnetic sector with rotating pole pieces to adjust the focal plane and a more compact design (lower volume means higher sensitivity) with greater spacing between the isotopes. It includes 5 individual multipliers, 4 of which are movable. Our own instrument, Supergnome M, uses a rotational focal plane for spacing adjustment, 7 independent multipliers and a high ( $\sim 100\%$ ) transmission ion source. As an instrument optimized for Xe, the pressure non-linearity of the GS-61 high transmission ion source is not a problem since the quantity of Xe extracted from the Genesis collectors will be small. The ion sources used in the GVI and the Nu Instruments

are less efficient but offer better pressure linearity. Given the importance of measuring heavy noble gases in the Genesis samples, it remains to be seen which system will produce the best isotopic data for Kr and Xe, the multiple collector instruments with lower efficiency or the high transmission instruments with multiple or single collectors. We are currently in the process of comparing these different approaches to obtain the optimum precision for Xe and Kr. It is only through measurement with multiple instrument types that optimization can be made for the measurement of each of the Genesis noble gases. Each instrument will continue to evolve as modifications are made to improve its performance.

One important task in comparing noble gas measurements obtained from different instruments and comparing noble gas results obtained from different groups is a uniform set of calibration standards. We needed to do this for the intercomparisons among our different instruments, and we designed and built a number of portable calibration sources. Realizing the importance of interlaboratory calibration, we built five of these. Four will be used among our different instruments, and the fifth will be sent to Minneapolis, Zurich, and other laboratories doing noble gas measurements, proving a common standard through which interlaboratory bias can be eliminated.

### **Work Statement:**

During the analysis phase of the Genesis mission, our work will continue to concentrate on improvements in our mass spectrometry capabilities through refinement of each of our new instruments. This work will include:

- Continued refinement of our Supergnome M, evaluating the performance of multiple small electron multipliers against that of a single electron multiplier. We have recently obtained a newly designed, high performance, electron multiplier which will soon be installed to make these tests. We continue to test the performance of "Noblesse" from Nu Instruments, with software and hardware refinement before extracting the large areas of solar wind required for good heavy noble gas analyses. "Helix", the GVI instrument is yet to be installed by GVI, awaiting delivery of improved miniature electron multipliers. Installation is expected later this month. Such things as final multiplier selection, conversion dynode optimization and ion source refinement will continue as calibration samples are compared among these new instruments. It is therefore likely that improvements will continue along with the operational data we obtain from each of these new instruments.
- We have integrated our new high-speed large-area (4" x 6") rastering stage into the sample system of "Noblesse" for heavy noble gas extraction. The laser system itself is on wheels for use with the smaller x-y stage on our other instruments, which is all that is needed for measurement of the light noble gases, with scan areas determined by the specific task at hand. We will continue to refine the computer software, integrating control of the mass spectrometers, the extraction laser and the high-speed x-y stage.
- We have refined laser extraction techniques for large area thin film removal, for controlled depth excavation and extended our low blank techniques to the new large area

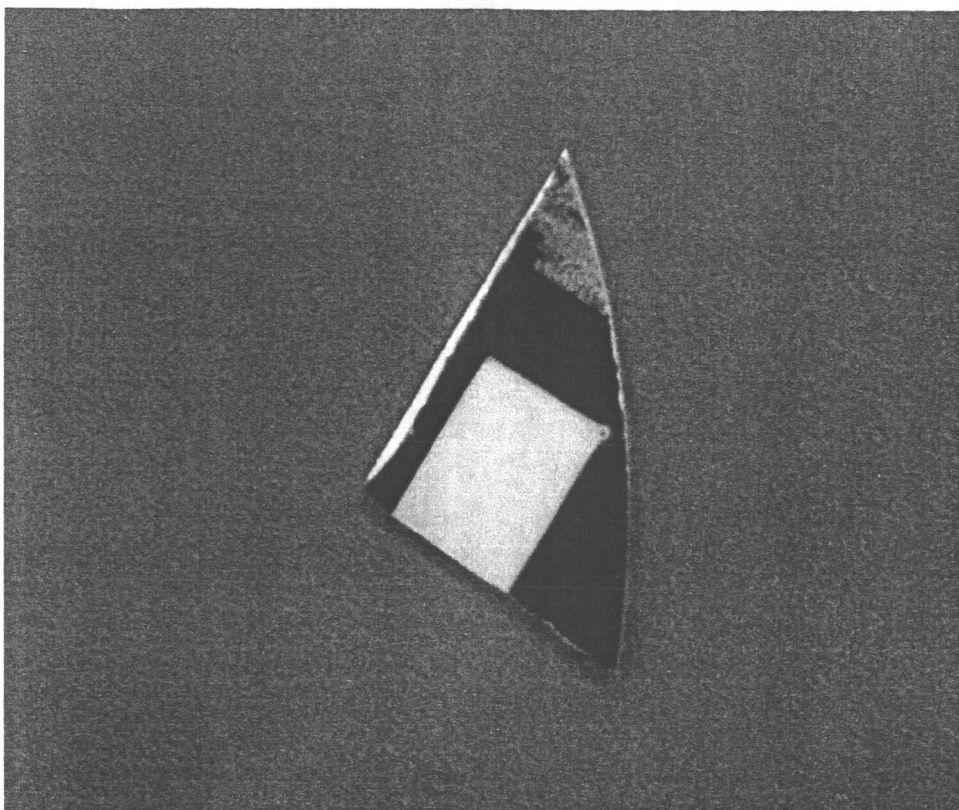
hardware. The problem presented here is the large amount of aluminum that is evaporated from the  $\sim 10 \text{ cm}^2$  areas needed for the heavy noble gases extracted from the aluminum "kidney", when compared with that removed from the thin films on sapphire substrates returned by the nominal (soft landing) mission. In order to prevent this aluminum from coating the sapphire laser entrance ports (coupling to the laser and destroying the ports), we have had to develop a moving mask that intercepts most of the evaporated aluminum. The first tests of this will be done shortly when blanks from (non-flight) kidney material are measured. Although the nominal blanks from our laser extraction cell are small, the added complexity of the moving mask may add to the blank which must be kept low due to the low abundance of solar wind Kr and Xe. The task at hand is to evaluate our operational blanks before measurement of Kr and Xe in the actual Genesis "kidney". Further refinement of these operational procedures will be an ongoing process, but the scarcity of material suitable for Kr and Xe measurements urges us to get it right before we commit to the measurement on the actual Genesis kidney.

- Procedural blank measurements of actual flight collector materials is under way. We will continue to strive to reduce the blank to optimize the precision and accuracy for the heavy noble gases.
- Measurement of all noble gases on structural components of the Genesis collector array, starting with the aluminum kidneys, will begin once we have optimized and verified our measurement capabilities including comparisons between each of our new instruments.
- Measurement of noble gases from the aluminum kidney material as a function of depth using controlled laser excavation.
- Measurement of noble gases from the Genesis collectors themselves using laser ablation. Differences between the different solar wind regimes will be addressed.

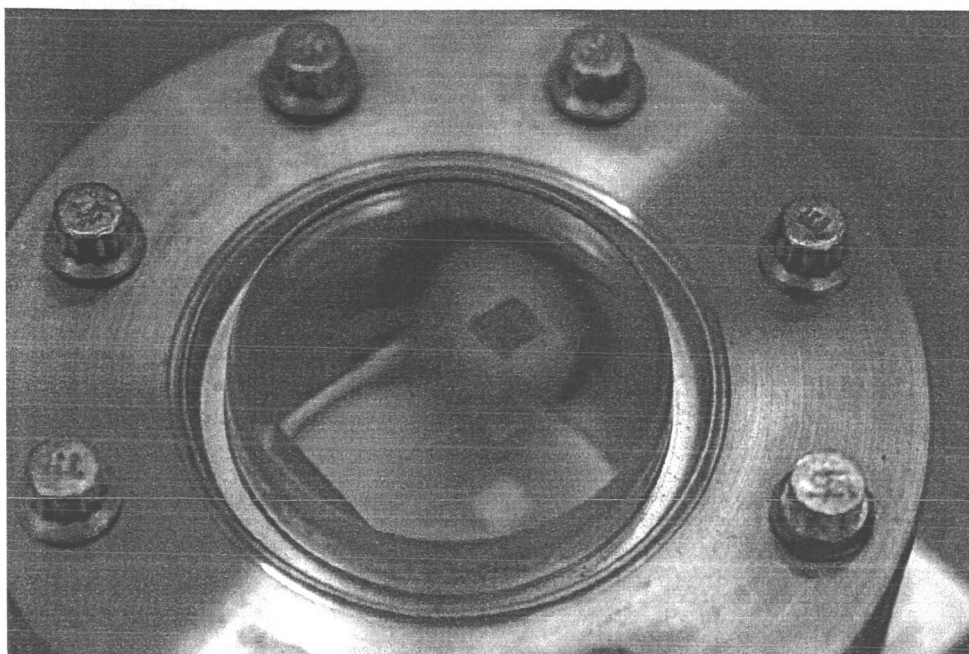
Figures 1 and 2 below shows the rastered area where the metal surface film containing the solar wind has been removed from a piece of sapphire substrate by laser ablation. The rumpled aluminum kidney material is not flat like the collector wafers and requires a long focal length lens and deeper extraction cell for surface removal. Also, more aluminum is removed the solid aluminum kidney than represented in the thin metallic films on the sapphire collector wafer, necessitating a movable shutter assembly to keep the evaporated aluminum from coating the sapphire entrance port (film can be seen in Fig. 2).

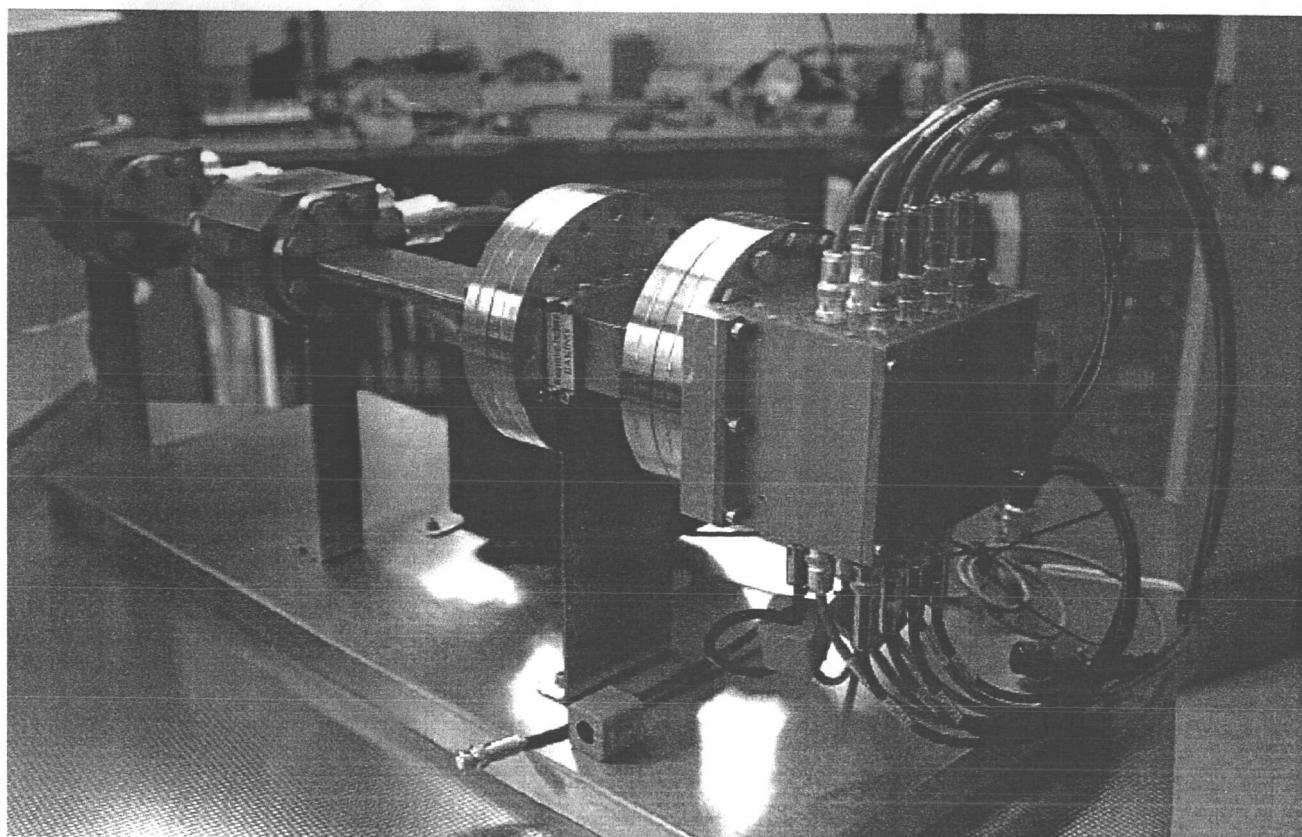
Figure 3 and 4 show Noblesse, the multiple multiplier mass spectrometer built in collaboration with Nu Instruments. Figure 5 shows Helix, built in collaboration with GVI, which has yet to be installed by GVI. Figure 6 shows Supergnome M built in house, based upon our current design with the high transmission GS-61 ion source.





Figures 1 and 2: Aluminum on sapphire collector where the surface film containing the Genesis noble gases has been removed by rastering with a UV pulsed laser. The actual flight material studied to date is the solid aluminum kidney. More aluminum is removed (see coating on sapphire port, Figure 2 below) , necessitating a movable shutter assemble to protect the sapphire view port through which the focused laser enters. The irregular surface of the crumpled kidney requires a longer focal length lens and taller extraction cell.





Figures 3 and 4: Noblesse, built in collaboration with Nu Instruments. This instrument has 8 fixed electron multipliers. Isotope spacing is changed by means of dual quad electrostatic lenses, shown in Figure 4 (bottom).

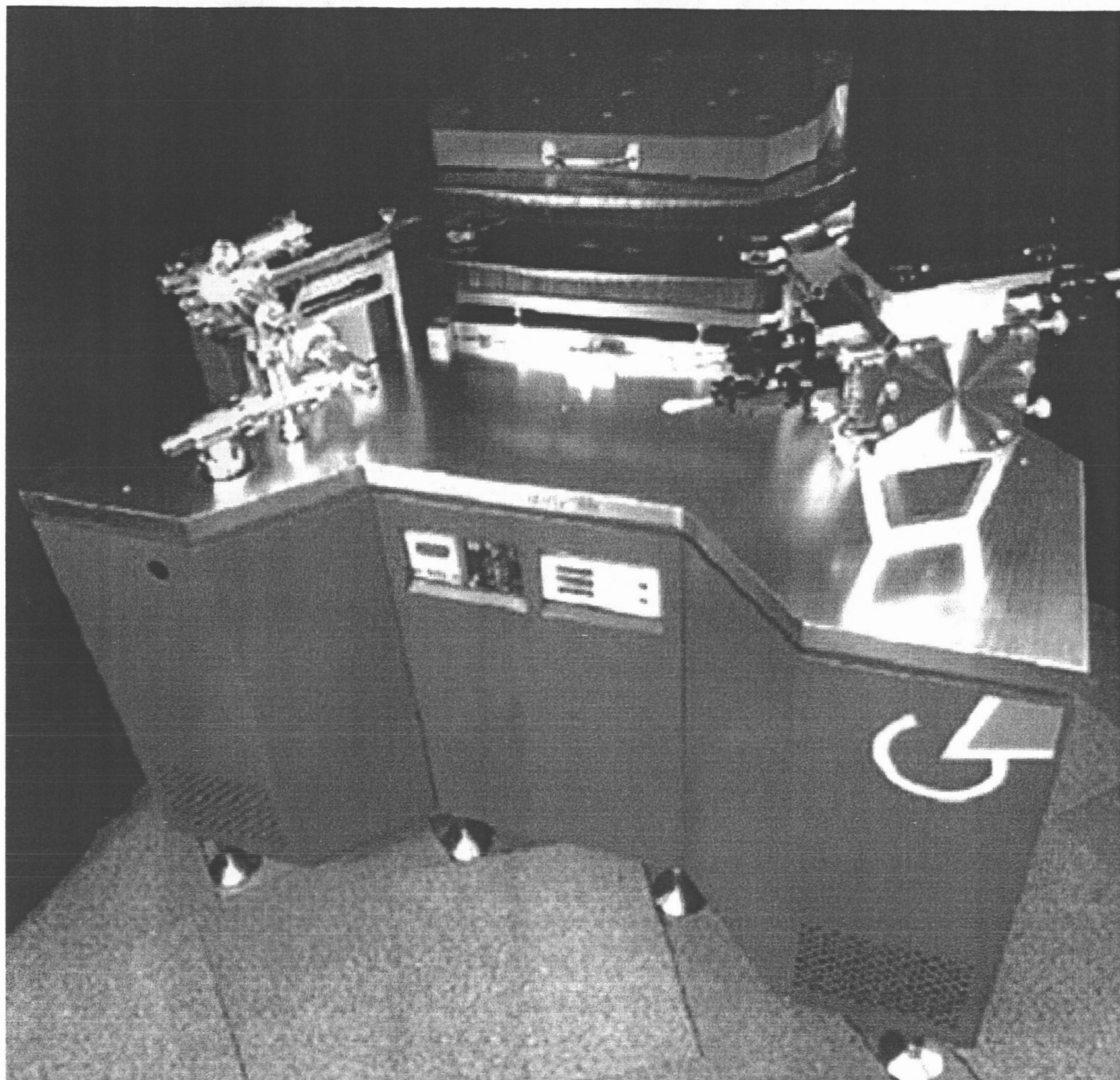


Figure 5: Helix, designed in collaboration with GV Instruments, as it sits in our laboratory. Helix has 5 individual channel electron multipliers, the center one is fixed and the two on each side are movable. Helix has not yet been installed by GVI due to problems with the performance of the miniature electron multipliers.



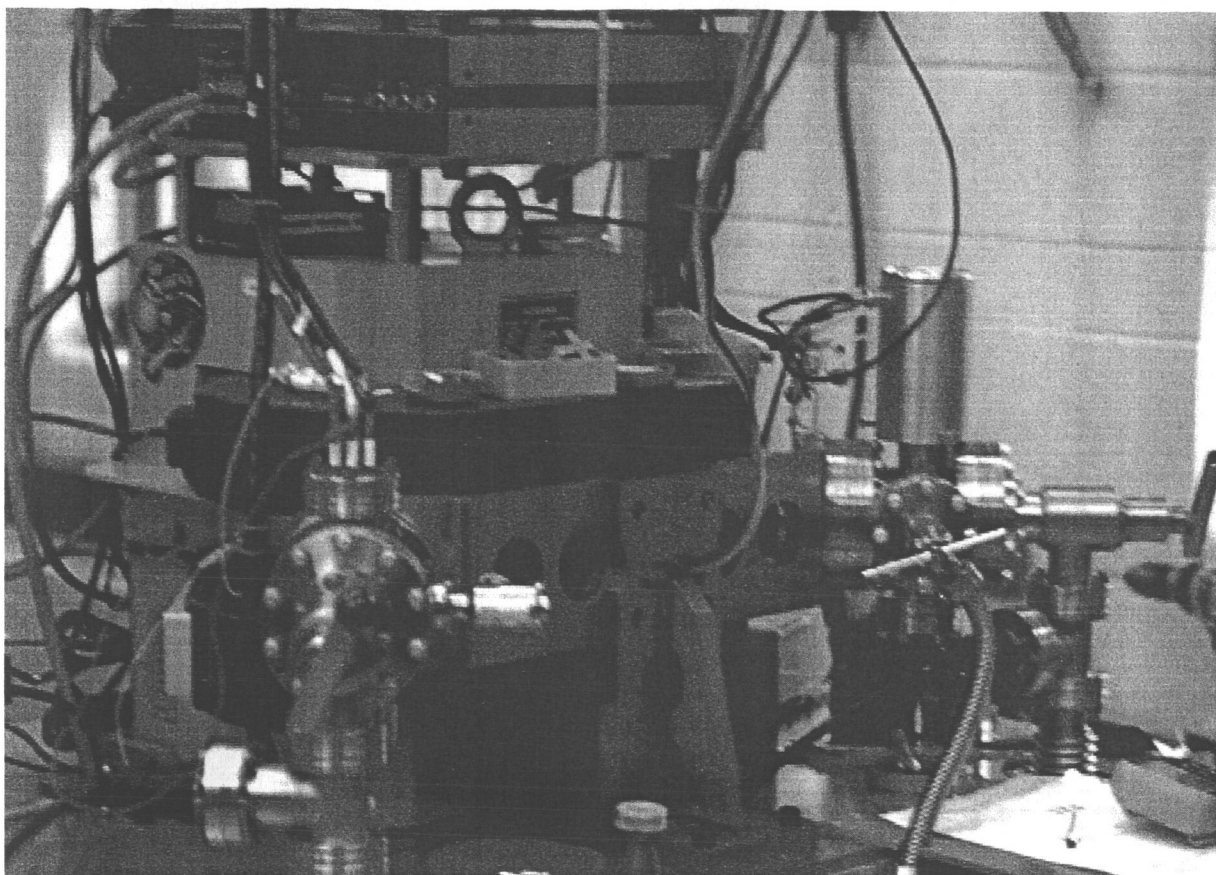


Figure 6: Supergnome M, built in house by our group, uses the same 90 degree normal entry magnetic sector and high transmission GS-61 ion source as our current mass spectrometers. The radius section of the flight tube was made by hydroforming from stainless steel, having no welds it has less magnetic heterogeneity than our current instruments. The hydroformed tube also maintains its width all the way to the collector and it has room for 7 miniature channel electron multipliers.